

Pyrolysis - Gas Chromatograph / Mass Spectrometer

- I. Instrument Name: Agilent 6890 Gas Chromatograph with Agilent 5975B Mass Selective Detector with CDS Analytical 1500 Manual Injection Pyrolysis Unit
- II. Suggested Uses: Paint, polymer, and organic analysis.
- III. Operating Procedures
 - A. Start-up and Calibration
 1. The GC-MS is kept on at all times.
 2. Calibration of the MS is done daily when in use with the Autotune program. Calibration procedures can be found in the Trace Evidence Section Calibrations Manual.
 3. Calibration of the GC will be verified monthly. Calibration procedures can be found in the Trace Evidence Section Calibrations Manual.
 - B. Collection and Storage of Data
 1. Prepare for sample injection
 - a. In the CDS 2000PLUS window, load the fbipyro program.
 - (1) Click on **File** and then click **Open**.
 - (2) Select the fbipyro method and click **open**.
 - (3) A box will come up asking "send to instrument?"
 - (4) Click **yes**.
 - b. In the Pyro 5975 window, load the pyrolysis35min method
 - (1) Click on **Method** and then click **Load Method**.
 - (2) Select the Pyrolysis35min method and click **OK**.
 - c. In the Pyro 5975 window, click on the sample vial.
 - (1) Load appropriate data folder
 - (2) Enter sample information into the spaces provided.
 - (3) Click **Start Run**.
 2. Cleaning Blank Tubes
 - a. Always wear white gloves
 - b. Place a tube containing only quartz wool into the probe, wool end down.
 - c. Unscrew the silver nut on the front of the pyrolysis unit.
 - d. Place probe into pyrolysis chamber and hand tighten the screw on the probe.
 - e. In the CDS 2000PLUS window, Click on the **Clean** button. Do this 3-4 times.
 3. Running Blank Samples
 - a. Press "Prep Run" button on front of GC
 - b. Wait 30 seconds.

- c. Turn the toggle switch on the pyrolysis unit to **RUN**.
 - d. Wait 10 seconds.
 - e. Simultaneously press the "START" button on the front of the GC and click on the "START/STOP" button in the CDS 2000PLUS window.
 - f. When the counter in the Pyro 5975 window reaches 1.00, turn the toggle switch on the pyrolysis unit to **LOAD**.
 - g. Remove the probe and place into the holding area.
 - h. Replace the silver nut on the front of the pyrolysis unit.
4. Samples
- a. Always wear white gloves.
 - b. Remove probe from holding area and carefully add the sample to the tube that is already in the probe.
 - c. Press "Prep Run" button on front of GC
 - d. Unscrew the silver nut on the front of the pyrolysis unit.
 - e. Place probe into pyrolysis chamber and hand tighten the screw on the probe.
 - f. Wait 30 seconds.
 - g. Turn the toggle switch on the pyrolysis unit to **RUN**.
 - h. Wait 10 seconds.
 - i. Simultaneously press the "START" button on the front of the GC and click on the "START/STOP" button in the CDS 2000PLUS window.
 - j. When the counter in the Pyro 5975 window reaches 1.00, turn the toggle switch on the pyrolysis unit to **LOAD**.
 - k. Remove the probe and place into the holding area.
 - l. Replace the silver nut on the front of the pyrolysis unit.
 - m. Remove tube from probe.
5. Bake Cycle
- a. After each sample, a bake cycle must be run
 - b. In the Pyro 5975 window, click on **Method** and then click **Load Method**. Select the "bake" method. Click **OK**.
 - c. In the Pyro 5975 window, click on the sample vial.
 - (1) Load appropriate data folder
 - (2) Enter sample information into the spaces provided (name sample "bake").
 - (3) Click **Start Run**.
 - d. Press the **Prep Run** button on the front of the GC.
 - e. When the "Not Ready" light on the front of the GC goes off, press the **Start** button on the front of the GC.
- C. Shut-down Procedures
- 1. Allow the oven to cool to approximately 50 degrees C.
 - 2. **DO NOT TURN THE GC-MS OFF!**

IV. Safety Concerns

- A. The probe gets very hot and can cause burns.
- B. The pyrolysis unit gets very hot and can cause burns.
- C. The injector and column areas are hot and can cause burns.

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